

Micromachines for Microchips: Bringing the AFM up to Speed

September 25, 2000
Final Scientific and Technical Report

Sponsored by:
Defense Advanced Research Projects Agency (DoD)
(Controlling DARPA Office)
ARPA Order K475/02

Issued by:
U.S. Army Aviation and Missile Command Under
Contract No. DAAH01-00-C-R014 / P00002
Contract Effective Date: October 26, 1999
Interim Option Effective Date: May 17, 2000
Interim Option Expiration Date: November 17, 2000

NanoDevices Inc., 516 E. Gutierrez, Suite E., Santa Barbara, CA 93103
805-884-0240, 805-884-0540 (Fax), steve@ndevices.com

PI: Stephen C. Minne, Ph.D.

The views and conclusions contained in this document are those of the authors and should not be interpreted as representing the official policies, either express or implied, of the Defense Advanced Research Projects Agency or the U. S. Government.

Approved for public release; distribution unlimited

DTIC QUALITY INSPECTED 4

20001010 111

REPORT DOCUMENTATION PAGEForm Approved
OMB No. 0704-0188

Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing this collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden to Department of Defense, Washington Headquarters Services, Directorate for Information Operations and Reports (0704-0188), 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302. Respondents should be aware that notwithstanding any other provision of law, no person shall be subject to any penalty for failing to comply with a collection of information if it does not display a currently valid OMB control number. **PLEASE DO NOT RETURN YOUR FORM TO THE ABOVE ADDRESS.**

1. REPORT DATE (DD-MM-YYYY) 25-09-2000		2. REPORT TYPE Final Report		3. DATES COVERED (From - To) May 2000 - September 2000	
4. TITLE AND SUBTITLE Micromachines for Microchips: Bringing the AFM up to Speed				5a. CONTRACT NUMBER DAAH01-00-C-R014	
				5b. GRANT NUMBER PAN RTW X4-00	
				5c. PROGRAM ELEMENT NUMBER	
6. AUTHOR(S) Minne, Stephen, C, Ph.D				5d. PROJECT NUMBER	
				5e. TASK NUMBER A001	
				5f. WORK UNIT NUMBER	
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) NanoDevices, Inc 516 E. Gutierrez, Suite E Santa Barbara, CA 93103				8. PERFORMING ORGANIZATION REPORT NUMBER	
9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES) Defense Advanced Research Projects Agency				10. SPONSOR/MONITOR'S ACRONYM(S) AMSAM-RD-WS-DP-SB	
				11. SPONSOR/MONITOR'S REPORT NUMBER(S)	
12. DISTRIBUTION / AVAILABILITY STATEMENT Distribution Unlimited					
13. SUPPLEMENTARY NOTES					
14. ABSTRACT This Interim Phase I / Phase II SBIR final report, developed under contract for topic number DARPA SB992-039, outlines improvements made to the atomic force microscope (AFM) in order to increase its imaging speed 10 to 100 times during standard operation. Faster imaging will allow the AFM to be used in high throughput military and microelectronic manufacturing applications. In the Phase I effort, the work done under this contract eliminated the rate-limiting element of the conventional AFM: the piezoelectric z-axis tube actuator. The interim work, which is described herein, developed a new type of tip for the AFM cantilever. This new tip is sharp, high aspect ratio, tall, and symmetric on the scan angle of the AFM. These are the necessary requirements for a cantilever that is to be used on the AFM system developed in the Phase I effort. With the new tip and the Phase I results, this effort is on track for completing the Phase II effort and moving the high speed AFM system into commercial and military application.					
15. SUBJECT TERMS Atomic Force Microscopy, High Speed Atomic Force Microscopy, AFM, Scanning Probe Microscopy					
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT UU	18. NUMBER OF PAGES 19	19a. NAME OF RESPONSIBLE PERSON Stephen C. Minne
a. REPORT U	b. ABSTRACT U	c. THIS PAGE U			19b. TELEPHONE NUMBER (include area code) 805-884-0240

Summary

The objective of this project is to develop an atomic force microscope (AFM) platform capable of high speed imaging in the contact and the intermittent contact modes of operation. The AFM is a unique tool for surface characterization because it is capable of producing three dimensional atomic scale images. As features on advanced electronics and data storage devices continue to shrink, obtaining high resolution measurements on these surfaces grows in importance. For these applications, traditional forms of metrology have been stretched to their limits. Standard optical microscopes are fundamentally limited by optical diffraction to around 100 nm. The scanning electron microscope (SEM) can provide nanometer scale resolution, but it does not produce three dimensional data. The AFM provides the necessary information with the necessary resolution, however it is not fast enough to be used in production applications. In the Phase I effort, this contract succeeded in increasing the throughput of the AFM by 30 times. In contact mode imaging this was accomplished by replacing the AFM's feedback actuator (an element which is normally the size of one's finger) with a micromachined device that is only a fraction of a millimeter long. For intermittent contact mode imaging, the speed was increased by using the previous technique coupled with an active filter that simulates viscous drag. The interim effort extended these results by building a AFM tip that is suitable for the high speed AFM. Unlike the tips previously worked with, the new tip is three times as tall (which reduces squeeze film damping), ten times sharper (which enhances horizontal resolution), and symmetric on the AFM's scan angle (which reduces tip/sample convolution and in some cases increases scan speed).

Table of Contents

Report Documentation Page, SF 298.....	1
Summary	2
Table of Contents	3
Introduction.....	4
Phase I Objectives.....	6
Overall Status & Worked Performed.....	7
Technical Description: The Ideal Tip	7
Part 1: Scan Angle and Symmetry.....	7
Part 2: Sharpness.....	12
Part 3: Tip Height.....	13
Part 4: Final Implementation	14
Estimates of Technical Feasibility.....	15
Future Plans	15
Contract Delivery Status	16
Report Prepared By.....	16
 Appendix I: Declaration of Technical Data Conformity	17
Appendix II: Distribution List	18
 Endnotes.....	19

Introduction

The ever-expanding military demands on the microelectronics, data storage, and biological industries require advancements in technology that are broad-based and crosscutting. Recently, the primary advances in these fields have come through system miniaturization. Advancements through miniaturization have placed a premium on high-resolution surface inspection and imaging. For many applications in these fields, the capabilities of traditional imaging techniques have been stretched to their limits. A robust high-speed high-resolution inspection tool is needed for continued miniaturization and enhanced functionality in these areas.

The atomic force microscope (AFM) was invented in 1986, and has since gained much popularity in high-resolution three-dimensional imaging. A schematic diagram of a typical AFM is shown in Figure 1. The AFM can be used in a wide variety of modes, including fluid imaging, magnetic imaging, capacitive imaging, and thermal imaging. However, the AFM has found its main use in high-resolution surface characterization. The two dominant AFM modes for surface characterization are the contact mode and the intermittent-contact mode.

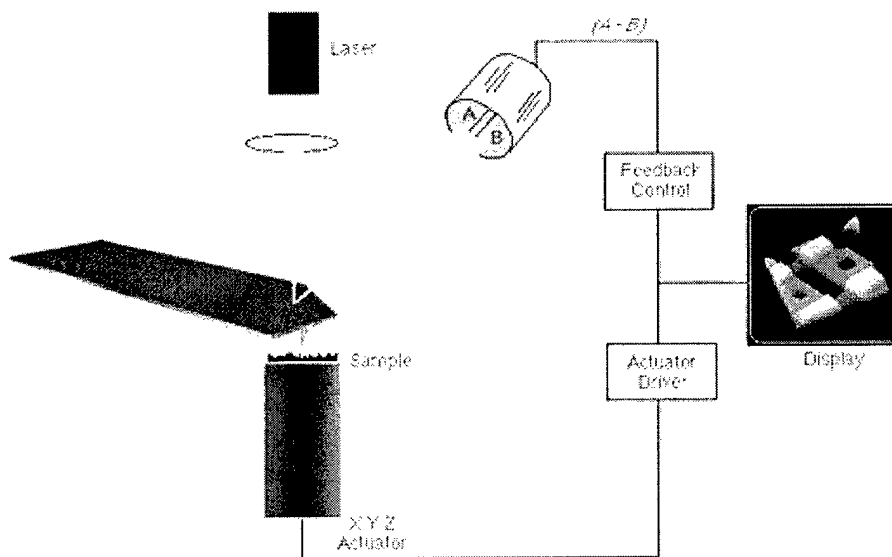


Figure 1: A schematic of a typical Scanning Probe Microscope (SPM). A image is formed by using the laser/cantilever/detector system to measure the height of every pixel of an image.

In the contact mode, the AFM operates by scanning an atomically sharp tip mounted on soft flexible cantilever across the surface to be imaged. As topographic features on the sample pass under the sharp tip, they cause the flexible cantilever to deflect. A sensor within the microscope monitors the deflection of the cantilever, recording the height of each pixel in the image. Since the raster scan is input by the user, the lateral position can be fed into a computer along with the measured height data, and a three-dimensional image of the surface can be rendered.

Typically, the contact mode AFM is operated in closed loop feedback. The feedback loop monitors the deflection of the cantilever and adjusts the sample position in

order to keep the forces between the tip and the sample constant. Maintaining a constant tip sample force preserves the delicate tip and protects fragile samples.

An intermittent contact mode microscope is very similar to the contact mode microscope. For intermittent contact imaging, two additional components are added to the schematic in Figure 1 (not shown). First, a small piezoelectric stack is placed under the cantilever, and second, an RMS to DC converter is placed after the split photodiode. During imaging, the cantilever is oscillated at its resonant frequency by the piezoelectric stack. As the tip rasters over the sample, the topographic data is obtained by measuring the degree to which the sample impedes the cantilever's oscillatory motion through the RMS to DC converter. Like in the contact system, feedback is used to move the sample relative to the tip to maintain a constant tip sample interaction. Otherwise the drivers, the electronics, and the actuators are the same as in the contact mode system. Intermittent contact mode imaging is the preferred method for AFM imaging because it eliminates lateral forces between the tip and the sample. (This mode of imaging is often referred to as Tapping Mode™, a trade name from Digital Instruments of Santa Barbara, CA.) Elimination of the lateral forces enhances the AFM's image fidelity and preserves the cantilever's tip sharpness.

It should be noted that the output of the AFM is not an exact image of the sample's surface. A more accurate description of the AFM's output would be a mathematical convolution of the sample's surface with the shape of the tip. In other words, if the tip is sharper than the features on the sample, the image will be dominated by the sample's surface. If the tip is duller than the features on the sample, the shape of the tip will dominate the image. This is true for features of all height. Figure 2 schematically depicts this relationship.

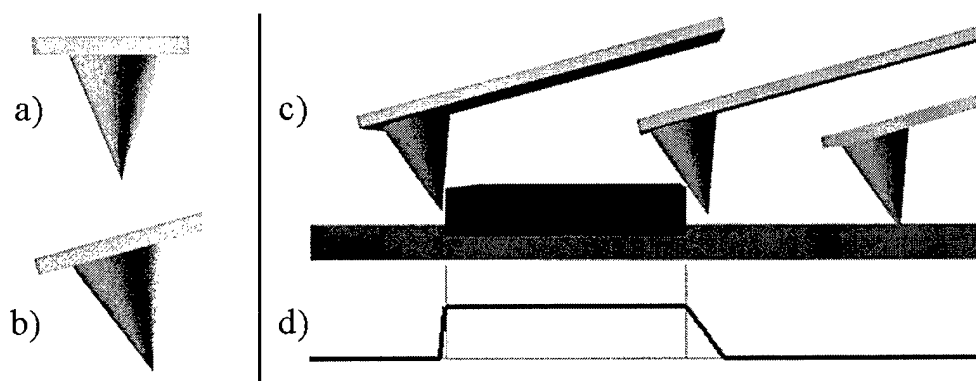


Figure 2: a) A typical tip defined perpendicular to the wafer, b) that same tip rotated to the scan angle of an AFM, c) the positions of the tip as it traverses a vertical step, d) the output of the microscope showing the convolution of the on scan angle tip shape and the sample.

An image typically consists of microscopic features and macroscopic features. The apex of the tip images the microscopic features, so to maximize the microscopic resolution; the tip must be very sharp (less than 10 nm). The sidewalls of the tip image the macroscopic features, so to maximize the image reconstruction accuracy; the tip's cone angle should be low and symmetric about the apex.

The tip shape also influences the ultimate imaging speed of the AFM. The connection between tip symmetry and speed can be explained from the tip sample convolution model. If both the tip and the sample have a steep edge, the convolution will also have a steep edge. Therefore, the higher harmonics in a feature's spatial frequency will be dependent on the angle of attack of the tip. Spatial frequency can be converted to bandwidth by multiplying by tip velocity. Since the bandwidth of the microscope is fixed, the maximum obtainable tip velocity is dependent on the angle of attack of the tip.

These different components of AFM horizontal resolution can be seen in Figure 3. Figure 3 is a line trace of a 3000Å overhanging step; a feature similar to that depicted in Figure 2. Figure 3 was taken using the most widely used commercially available cantilever – the NanoSensors PointProbe. The convolution effect from the sidewalls can be seen in the sloped lines on each side of the step. If the tip were ideal, these would be perfectly vertical lines. Asymmetry in the tip shows up in the left slope being different from the right. Clearly the image is both inaccurate and distorted. Less visible is the microscopic variation in on the flat surfaces. The radius-of-curvature of the tip limits the high frequency spatial resolution in these regions.

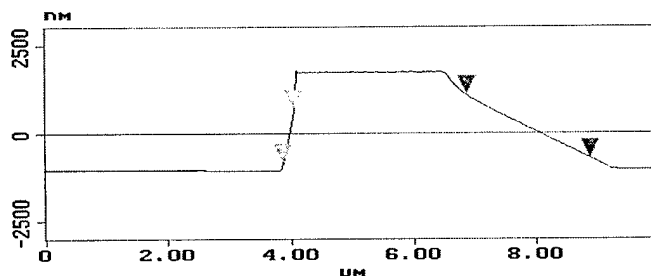


Figure 3: Output profile of a tip passing over an overhanging feature. The left side sidewall angle is 83 degrees and the right side sidewall angle is 41 degrees. The composite cone angle is 56 degrees. Note the asymmetry in the profile due to the scan angle.

Fabrication of an ideal AFM tip is a difficult problem – it must be symmetric “on scan angle”, high aspect ratio, and sharp. We propose to fabricate this ideal tip using anisotropic wet etching of silicon. An additional constraint of our process is that it must be compatible with the high speed cantilever fabrication (see Phase I reports and Phase II proposal for a complete description of the high speed system).

Phase I Objectives

The overall objective of the proposal was to develop a commercially viable system for very high speed atomic force microscopy in the contact and the intermittent contact mode. The system should be relevant to research, manufacturing, and production applications. During the interim contract, the effort was directed towards building a superior AFM cantilever tip that is compatible with the previously accomplished high speed AFM work.

The objectives of the Interim proposal were be enumerated as follows:

- 1) Design the masks for a tall sharp high-aspect ratio "on scan angle" symmetric tip.
- 2) Build and test the tip structure on bare silicon wafers.
- 3) Analyze the tip i) scan angle, ii) sharpness, iii) height, iv) symmetry, and v) footprint for suitability as an AFM tip.
- 4) Redesign in necessary, and incorporate the tip onto a 40um x 125um cantilever structure.

Overall Status & Work Performed

During this interim contract a new advanced AFM tip was developed. Unlike any other commercially available tip, this tip is very symmetric on the scan angle of the AFM. This has two main advantages: 1) the image's distortion from tip/sample convolution is minimized. 2) By eliminating any vertical sidewalls on the tip, the spatial frequencies that the AFM encounters to are reduced, thereby increasing the microscope's overall speed potential.

In addition to the benefits of the on scan angle symmetric tip, these tips are taller sharper, and have a smaller end cone angle than other available tips. The enhanced tip height reduces squeeze film damping, and improves intermittent-contact and non-contact imaging performance. The sharper apex improves the microscopic horizontal resolution of the AFM. The smaller cone angle improves the macroscopic horizontal resolution.

The new tip structure has been integrated onto a standard AFM cantilever. Images with the new cantilever with improved tip are presented. Details of the experimental configurations are described in the following sections. These results meet or exceed all of the solicited and proposed objectives.

Technical Description: The Ideal High Speed AFM Tip

Part 1: Scan Angle & Symmetry

The disposable nature of the AFM cantilever, coupled with its nano-scale properties, force effective fabrication to be MEMS based. Standard MEMS processing relies on tools in silicon wafer fabs. This causes tips to be generally created with dry etching, or molding of (111) silicon pits. These processes are inherently symmetric to the plane of the wafer. Unfortunately, in order to approach the sample surface, the AFM angles the cantilever die. This makes tips symmetric about the cantilever surface asymmetric on the operational scan angle. There is more versatility using anisotropic etching of silicon to make an "on scan angle" symmetric tip, however such tips are not available.

Using the anisotropic crystal etching simulator (ACES, shareware from the University of Illinois) we have found a process to make an ideal tip. Figure 4 shows the simulation output for a trapezoidal mask. The tip is tall, high aspect ratio, and binds the (411) silicon planes. The cone angle is 55 degrees divided down the center of the tip into 20 degrees and 35 degrees. This will produce "on scan angles" of 30 degrees (20 + 10)

and 25 degrees ($35 - 10$), or only a 5 degree asymmetry. This is a great improvement over conventional cantilevers, which can have attack angle differences as great as 40 degrees.

With this simulation, masks for fabricating tips were designed. Wafers were prepared with a silicon nitride film, and then the pattern was transferred into the film using standard silicon processing. The wafers were then etched in KOH until the silicon nitride mask was completely undercut. Figures 5 & 6 show SEM micrographs of the fabricated tip's front and side profiles respectively.

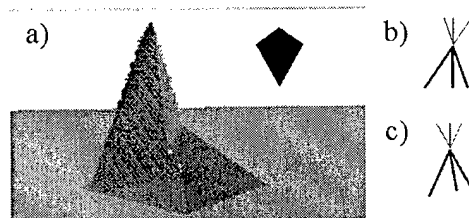


Figure 4: a) A simulation of a "on scan angle" symmetric tip using anisotropic etching of silicon. The inset shows the mask shape. b) Line traces through the x-axis profile of the tip. The vertical line is a normal to the cantilever surface. The scan cone angle is 55 degrees broken divided around the normal to 20 degrees and 35 degrees. The thin lines above the profile show the equivalent shape of the tip if the scan angle were 0 degrees. c) The profile of part b) rotated to a 10 degree scan angle. The thin lines now show the on scan angle tip shape. The vertical line is now a normal to the sample. The "on scan angle" tip shape is now a 55 degree cone split to 30 degrees and 25 degrees. The y-axis profile is symmetric with a cone angle of 30 degrees.

The on scan angle symmetry in these tips are better than any other commercially available tip. From Figure 6 it can be seen the included angle is 39 degrees, which is split about the normal into 15 degrees and 24 degrees. When the tip is inserted into the AFM, it is angled to 13 degrees.¹

With the 13 degree scan angle, the new effective tip shape, relative to the normal of the sample, becomes 28 degrees and 11 degrees. This improved symmetry can be seen in Figure 7. Figure 7 shows the profile of the newly developed tip compared to that of a conventional AFM tip (NanoSensors - PointProbe). The conventional tip is very asymmetric on scan angle. The leading edge impacts any step edge almost vertically (4 degrees from normal). The trailing edge is much too gradual (40 degrees). The effect of the trailing edge can be seen by the total width of the 1.5 μm step being extended to over 2.5 μm . The newly developed tip better represents the vertical step on the sample; the entire feature is extended to only 2.1 μm .

Figure 8 shows the tip profile in the orthogonal direction. The tip's included angle is 35 degrees, which provides good symmetry with the opposite direction (39 degrees). It should be noted that the entire cone angle is reduced to about 27 degrees at the top 1 μm of the tip. This reduction in cone angle is from an extensive oxidation process that both sharpens and smooths the tip shape. This cone angle reduction is not seen on other available tips.

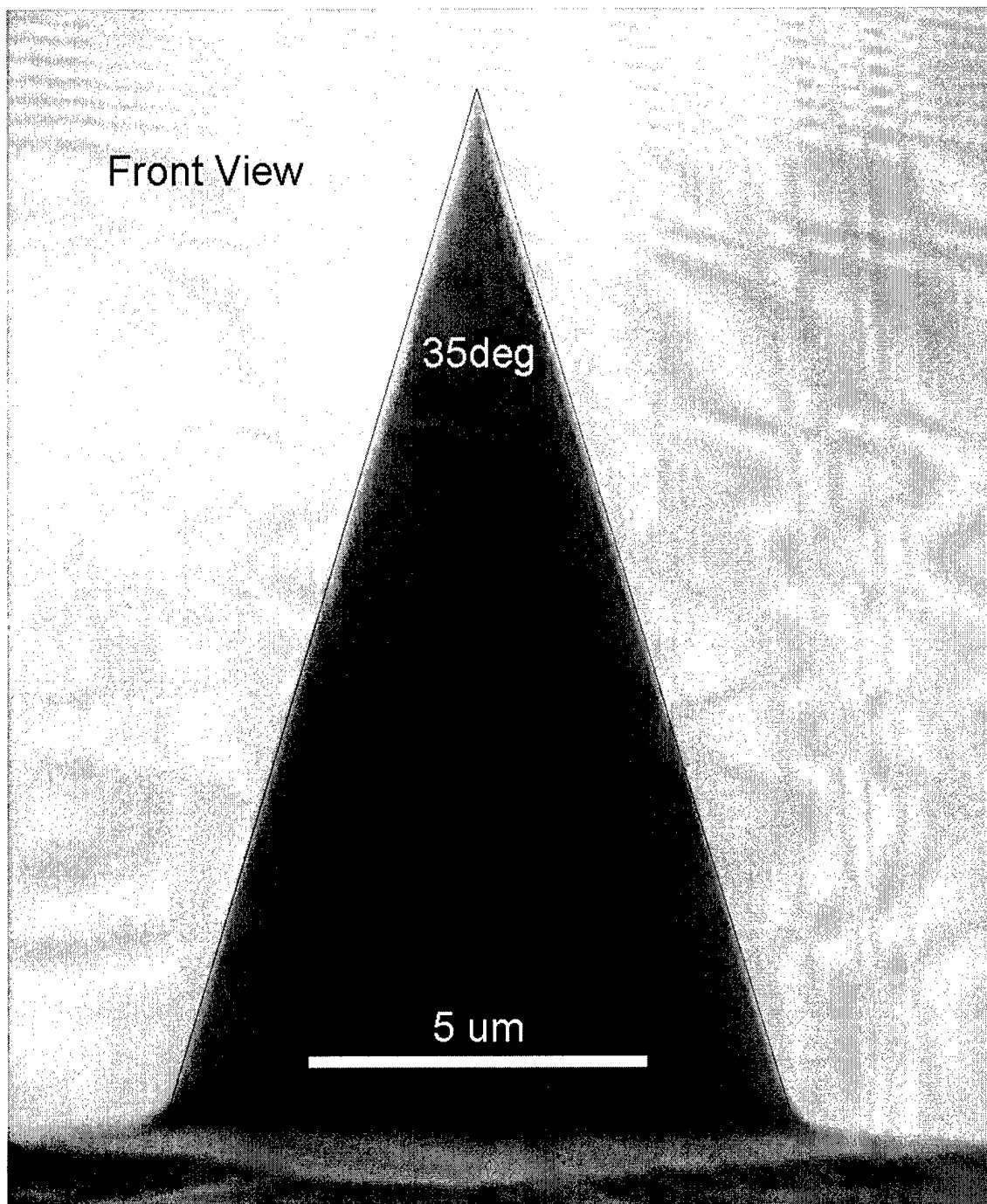


Figure 5: Front view of the tall, sharp, high aspect ratio, on scan angle symmetric tip. Typical tips are between 15um and 20um tall, have a end radius of curvature of less than 10nm and, a included angle of 35 degrees.

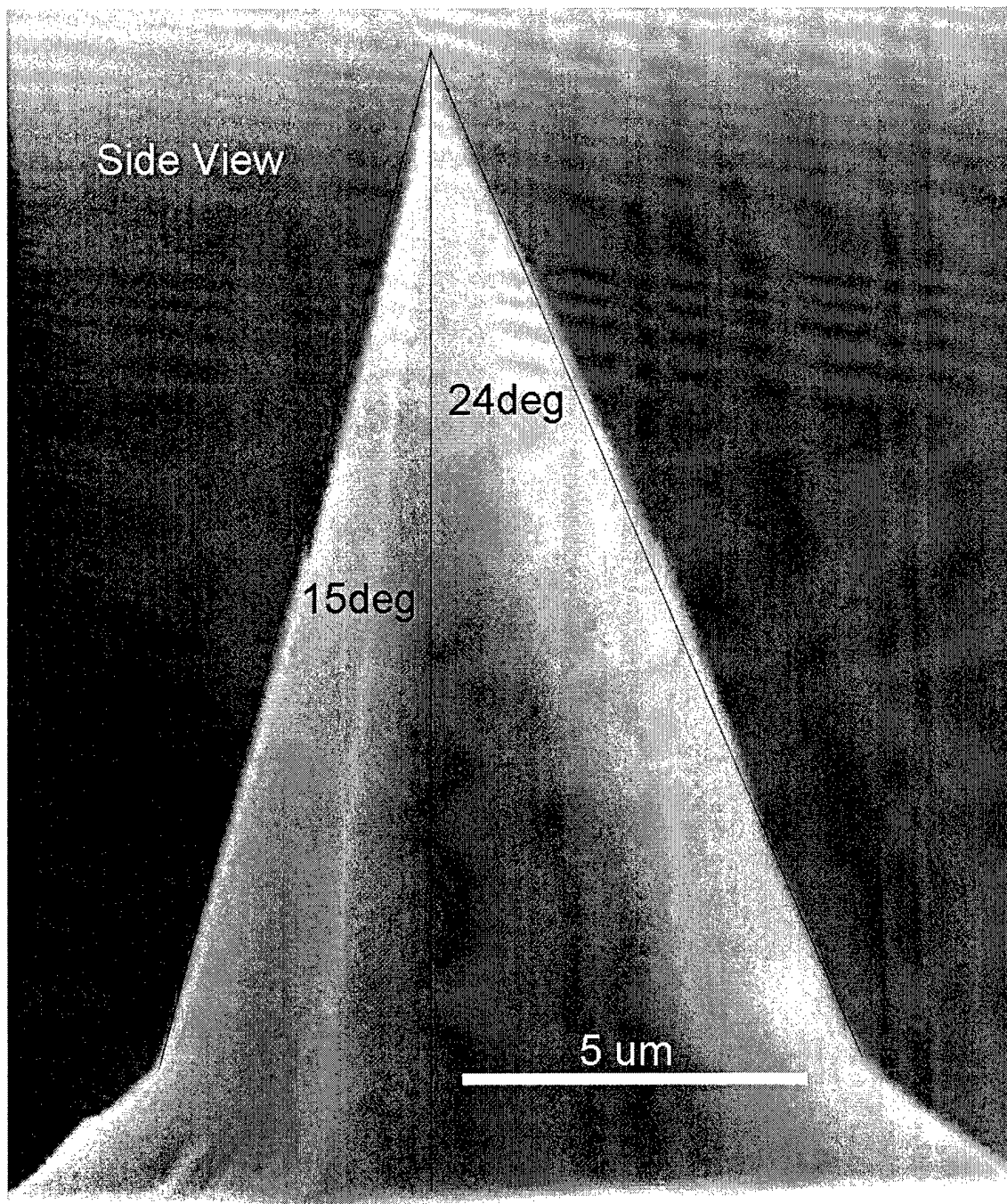


Figure 6: Side view of the tall, sharp, high aspect ratio, on scan angle symmetric tip. The side view total included is 39 degrees, split 15 degrees and 24 degrees around the normal. This asymmetry on the wafer better produces an "on scan angle" symmetry.

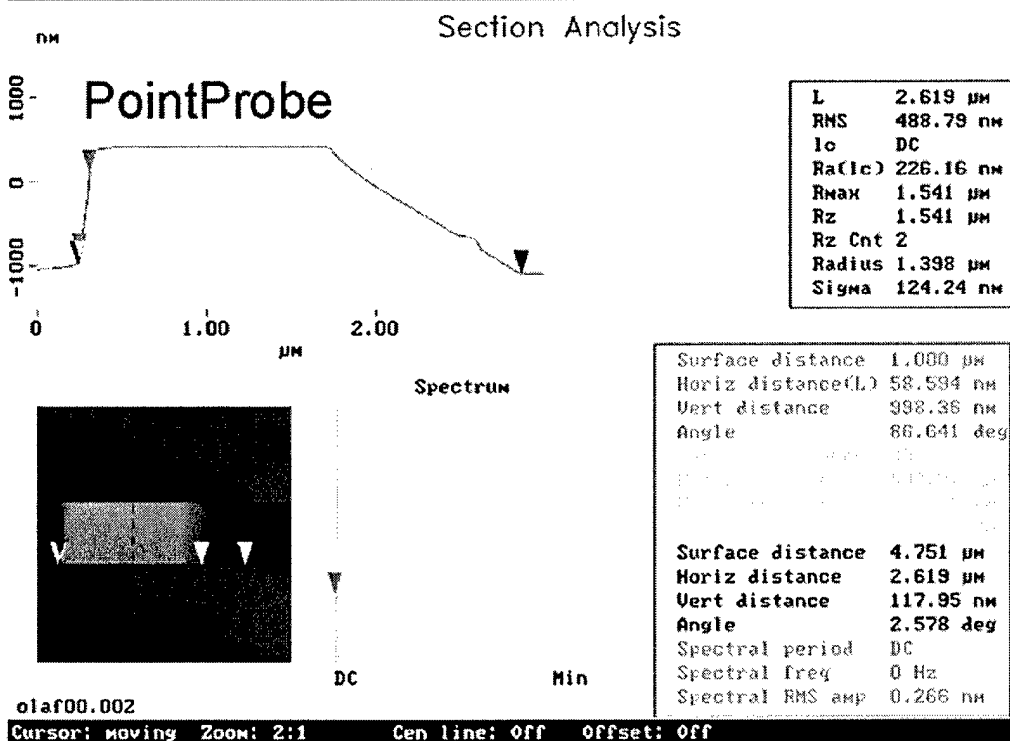
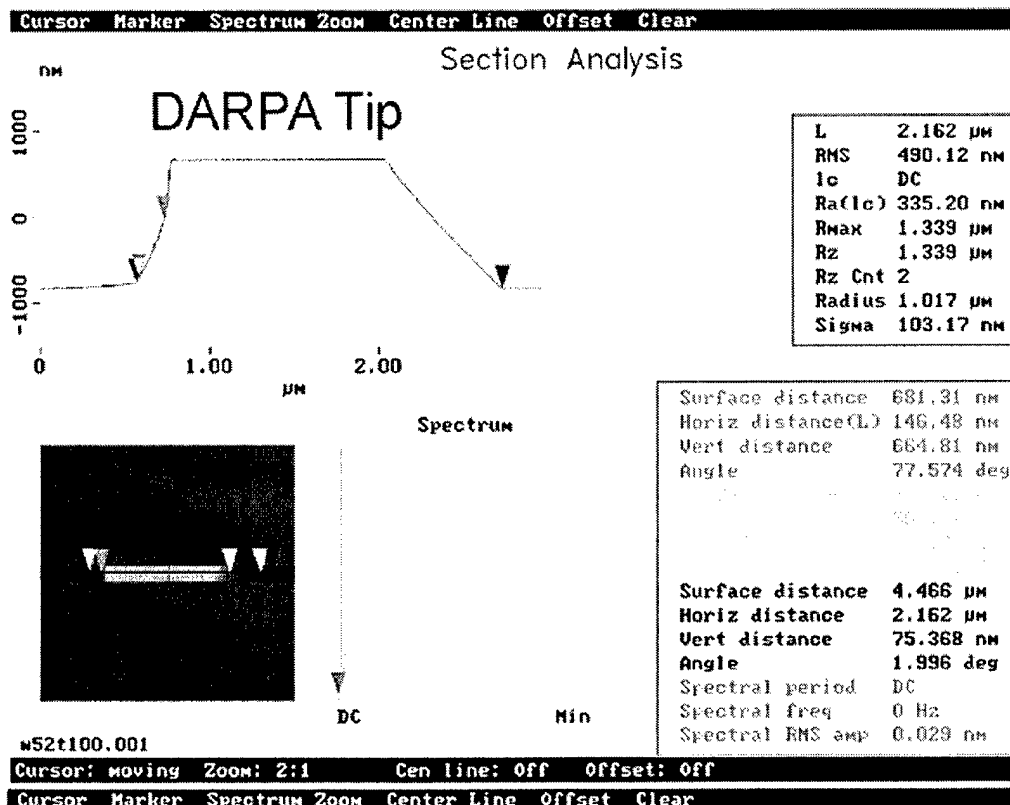


Figure 7: Comparison of side wall angle between (top) the newly developed tip and (bottom) the NanoSensor PointProbe, the most commonly used AFM tip. The new tip as considerably less asymmetry relative to the surface. This will allow for better image reconstruction and faster scanning.

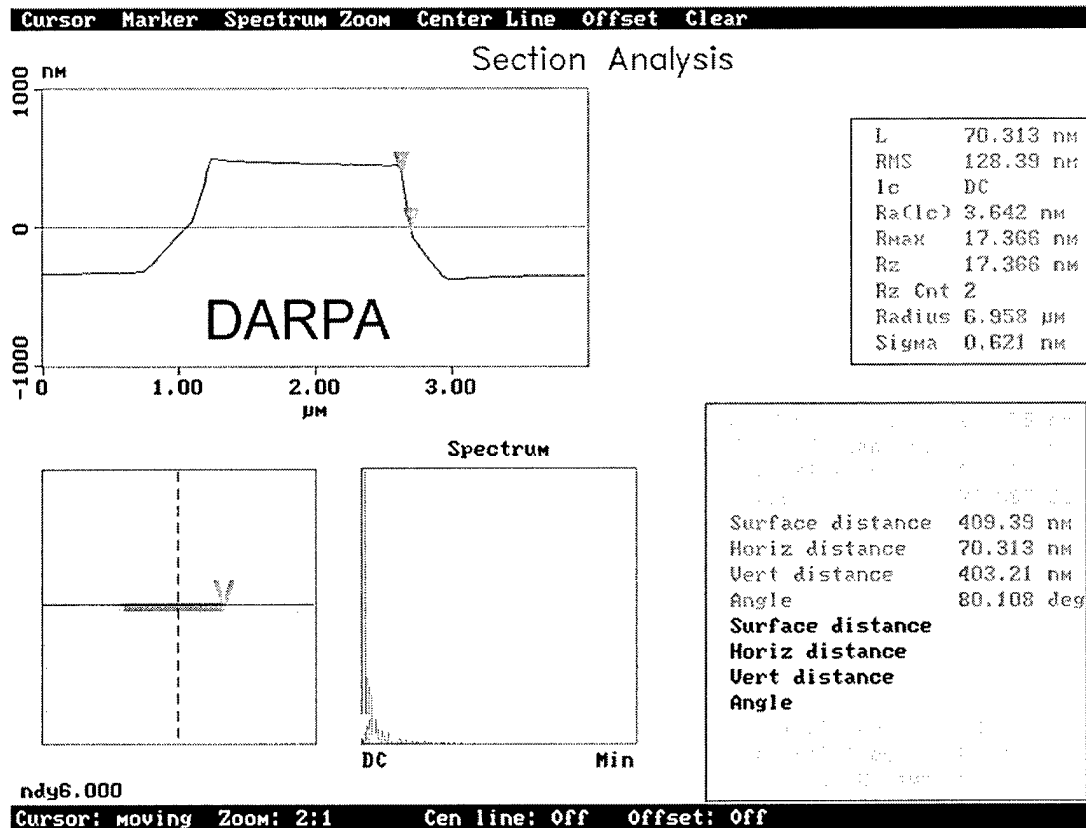


Figure 8: Shows the horizontal symmetry in the newly developed top. The markers in the graph show how extensive oxidation sharpening further reduce the total included angle of the tip.

Part 2: Sharpness:

The shape of the tip apex determines the microscopic horizontal resolution. This parameter is usually characterized by radius of curvature, and this radius determines the minimum feature that can be imaged by the AFM. There are many ways to measure the end radius of a tip. The most obvious, but most difficult, is a direct measurement with a transmission electron microscope. This type of characterization is expensive and time consuming, and cannot be done in real time for a given tip. A more common approach is to create an algorithm that calculates the maximum slope in every direction of an image and reconstructs the worst case tip. Characterization packages that provide the algorithm and a suitable sample are commercially available.

Figure 9 shows a mathematical characterization of the newly developed tip. Figure 9a is an image of a titanium sample with 100nm sharp features. This image is then analyzed and the points of maximum slope in every direction are found. In figure 9a, the small gray "plus signs" mark these points. From these points figures 9b and 9c are created. Figure 9b is the maximum possible cross section of the tip at 5nm from the apex. The tip has a radius of curvature of 8nm at this point. Figure 9c is the cross section at 10nm from the apex. At this height, the effective radius of curvature is 12 nm.

Typical curvatures of commercially available tips are 15 nm. The extensive oxidation sharpening used in the NanoDevices process provides a tip of exceptional sharpness.

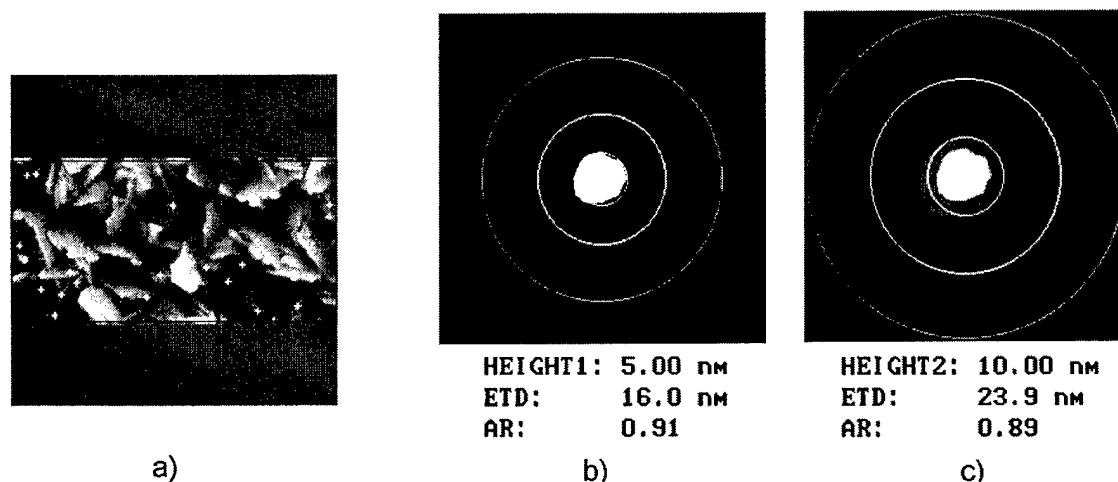


Figure 9: a) AFM image of a titanium sample containing numerous small sharp ridges. b) a mathematical model of the maximum sized tip that could have formed the image shown in a) at 5 nm up the tip. The estimated total diameter (ETD) is 16 nm and the aspect ratio (AR) 0.9. c) a mathematical model of the maximum sized tip that could have formed the image shown in a) at 10 nm up the tip. The ETD is 24 nm and the AR is 0.9.

Part 3: Tip Height

In tapping mode, the height of the tip also influences the imaging dynamics. A tall tip (greater than 10 μm) is needed to prevent squeeze film damping of the oscillation.² Tips shorter than this lead to unstable intermittent contact mode images because the oscillation amplitude becomes small and cannot be maintained over large features. Figure 10 is a series of tips, in both side and front view, which show we are able to produce repeatable tall sharp tip structures.

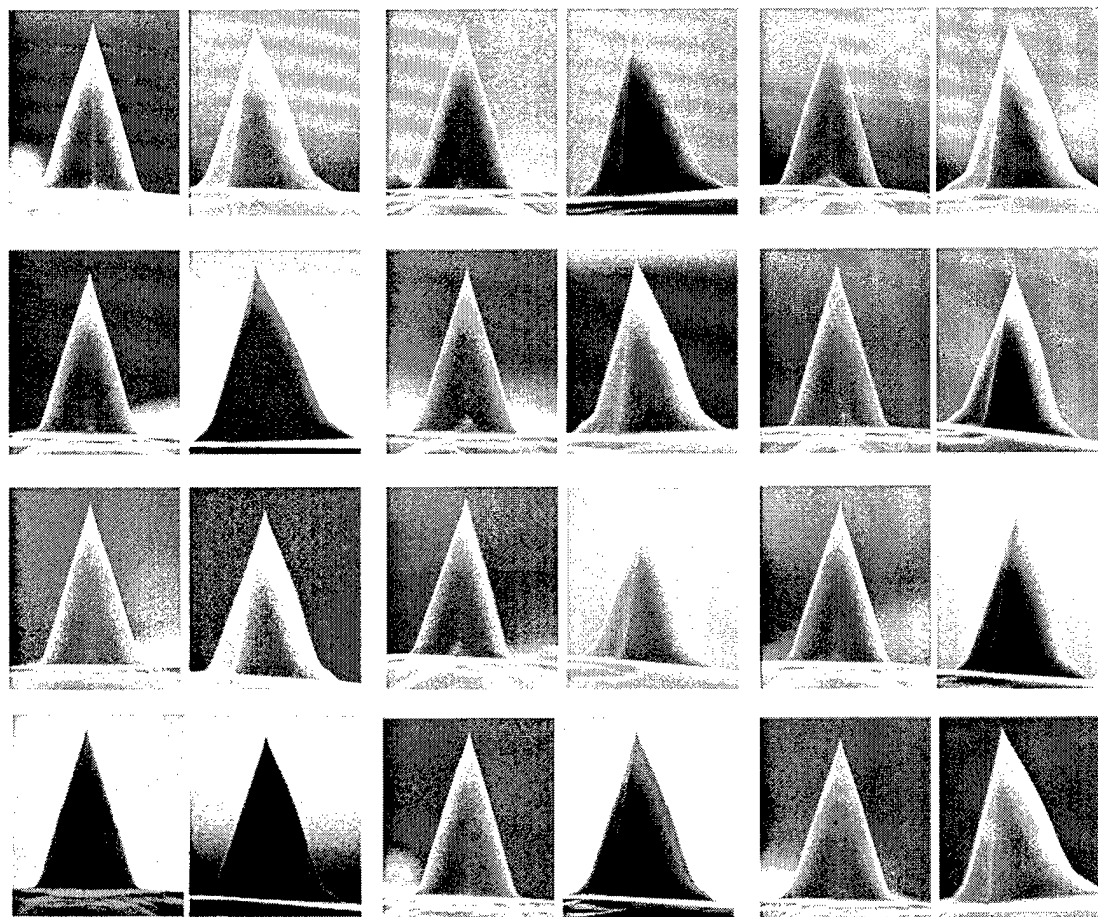


Figure 10: SEM micrographs of the newly developed tips showing tall uniform repeatable structures. The scale for all the images is 1/2 inch = 10 μm .

Part 4: Final Implementation

Once it was determined the new tip structure had exceptional probe properties, in addition to having a suitable footprint and process for integration onto a cantilever, it was incorporated onto a standard AFM cantilever. Figure 11 shows the final implementation of these tips onto AFM cantilevers. This device has a nominal resonant frequency of 300 kHz and nominal spring constant of 40 N/m. It is fabricated from (100) monolithic silicon (n-type, Phosphorous). The cantilever dimensions are 125 μm length, 45 μm width, and 4 μm thickness. Typical Q values in air are around 300.

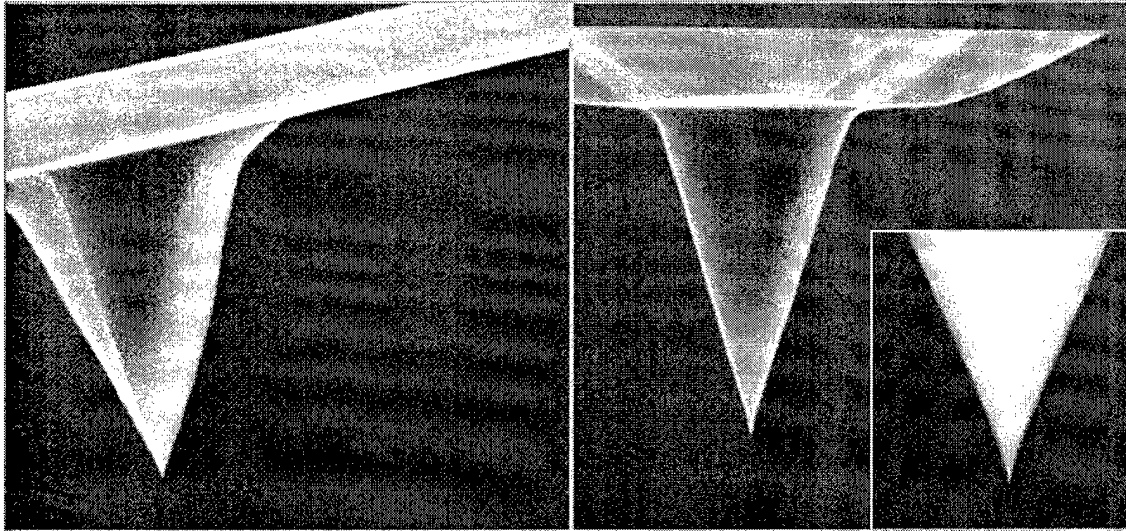


Figure 11: Final implementation of the newly developed tip onto an AFM cantilever. The side view of the cantilever (left) is angled to the scan angle of the AFM. The symmetric scan profile is clearly seen. The front view (right) and the detail of the front view (right inset) show the sharpness and cone angle tapering of the tip. For scale, the cantilever shown is 4 μm thick.

Estimates of Technical Feasibility

The research carried out under this contract provides a sound base for the commercialization of a very high-speed atomic force microscope based on a MEMS cantilever with an integrated actuator, custom tip, and specialized circuitry. Typical microscopes scan at tens to hundreds of microns per second. In previous work under this contract we have demonstrated scan speeds of up to a centimeter per second. This speed advance changes the whole look and feel of the AFM from a research tool that takes hours of use to obtain final data, to a tool that images in real time with scan and zoom capabilities. This will both change the how the AFM is perceived for high throughput manufacturing applications as well as open new doors for short time scale research applications. The high speed AFM is technically feasible. Further research is needed to understand the effects of high speed imaging on resolution, linearity, and tip wear. These topics will be the focus of the Phase II proposal.

Future Plans

We plan to continue to advance the state of the art in high-speed atomic force microscopy through further cantilever, microscope, and electronic innovation. Our Phase II Fast Track proposal has been accepted and we prepared to begin work as soon as the contract is finalized.

Contract Delivery Status

There is one deliverable on this contract: This interim statement of work. Due November 17, 2000; submitted September 25, 2000; on time.

Report Prepared By

Stephen C. Minne, Ph.D.
NanoDevices Inc.
516 E. Gutierrez, Suite E
Santa Barbara, CA 93103
805-884-0240
805-884-0540 (fax)
steve@ndevices.com

Appendix I:

Declaration of Technical Data Conformity

The contractor, NanoDevices Inc., hereby declares that, to the best of its knowledge and belief, the technical data delivered herewith under contract No. DAAH01-00-C-R014 is complete, accurate, and complies with all requirements of the contract.

Stephen C. Minne, Ph.D.
President, NanoDevices, Inc.

Date

Appendix II: Distribution List

Commander
U.S. Army Aviation and Missile Command
ATTN: AMSAM-RD-WS-DP-SB
(Mr. Alexander H. Roach, Technical Monitor)
Bldg. 7804, Room 205
Redstone Arsenal, AL 35898

2 Copies

Commander
U.S. Army Aviation and Missile Command
ATTN: AMSAM-RD-OB-R
Bldg. 4484, Room 204
Redstone Arsenal, AL 35898-5241

1 Copy

Commander
U.S. Army Aviation and Missile Command
ATTN: AMSAM-RD-WS
Bldg. 7804, Room 247
Redstone Arsenal, AL 35898-5248

1 Copy

Director
Defense Advanced Research Projects Agency
ATTN: MTO (Dr. Christie R. K. Marrian)
3701 North Fairfax Drive
Arlington, VA 22203-1714

1 Copy

Director
Defense Advanced Research Projects Agency
ATTN: ASBD/DARPA
3701 North Fairfax Drive
Arlington, VA 22203-1714

1 Copy

Director
Defense Advanced Research Projects Agency
ATTN: ASBD/DARPA Library
3701 North Fairfax Drive
Arlington, VA 22203-1714

1 Copy

Defense Technical Information Center
ATTN: Acquisitions/DTIC-OCF, Rm-815
8725 John J. Kingman Rd., STE 0944
Ft. Belvoir, VA 22060-6218

2 Copy

Endnotes

¹ This angle allows the AFM to approach the tip to the surface without engaging the entire cantilever die and/or mount.

² F. M. Serry, P. Veuzil, R. Vilasuso, and G. J. Maclay, in *Proceedings of the Second International Symposium on Microstructures and Microfabricated Systems*, Chicago (Electrochem. Soc., Pennington, NJ, 1995), p.83.